4. EXPERIMENTAL DESIGN AND PROCEDURES

4.1 Data Quality Objectives

The seven-step Data Quality Objectives (DQO) process (U.S. EPA 1994) was employed to develop test plan quality objectives. A summary table of the DQOs is given in Appendix A. The data quality requirements are based on the ability of the CFS system to stabilize the RCRA metals. This includes soils from the sites shown in Table 3. Ultimately, the treatability study will be used to determine whether stabilized waste can meet the acceptance criteria of the ICDF. For the treatability study testing, the analyses of the stabilized surrogate and untreated, spiked samples, will consist of:

- Paint Filter Test
- TCLP for RCRA metals.

4.2 Test Design

4.2.1 Introduction

This section will describe the number of samples, number of recipes, and the number of test iterations that will be conducted. The test design and strategy will attempt to minimize sampling and analysis, while providing a minimal number of CFS formulations. The strategy is to conduct tests on surrogate wastes prepared from representative soils to determine CFS formulations that bind all contaminants of concern. The surrogate testing will provide a baseline CFS formulation and starting waste loading for verification testing.

4.2.2 CFS Chemistry

Solidification processes use chemically reactive formulations that, together with water and other components, form stable solids. Stable, in this case, means that the solids are physically stable under normal or expected environmental conditions and will not revert to the original liquid, semi-liquid, or unstable solid state (Conner 1990).

Chemical fixation and solidification systems not only solidify the waste by chemical means, but also insolubilize, immobilize, encapsulate, destroy, sorb, or otherwise interact with selected waste components. The purpose of these systems is to produce solids that are nonhazardous, or less hazardous, than the original waste. The goal for the SSSTF soil stabilization is to sorb, insolubilize, and immobilize the metals Ag, Hg, Cd, Cr, and Pb. This will be referred to as fixation and stabilization.

Treatment formulations for this work will include one or more of the following reagents: Portland cement Type I (PC), blast furnace slag (BFS), Class F flyash, and sodium sulfide. Various combinations of these materials will be examined to determine suitable formulations to meet the stated treatment criteria.

Table 3. Potential wastes for treatment (concentrations are total sample analyses).

Release Site	Vol. (yd^3)	Matrices	Cd (mg/kg)	Cr (mg/kg)	Pb (mg/kg)	Hg (mg/kg)	Ag (mg/kg)	Organic (mg/kg)	PCB (mg/kg)
ARA-12	2,000	Sandy, silty clay with rock pieces[a]	24	460	158	1.4	300		
Borax-01	11,110	Imported gravel in an area of silty clay soil[b]	120	940	3,340	5.4	2	104.7	1.24
CFA-04	800	Rocky soil with a small percentage of calcine[c]	6	240	40	440	122	0.001	3.18
CPP-92	1,197	Soil[d]							
	4	Metal[d]				20			
	116	Concrete[d]							
	53	Soil/Asphalt/Concrete[d]							
CPP-98	30	Soil[d]							
	209	Wood/Nails/Bolts[d]							
	7	Metal[d]							
	4	Undetermined[d]							
CPP-99	30	Soil[d]							
	2	Wood/Nails/Bolts[d]							
	11	Metal[d]							
	62	Concrete[d]							
	12	Soil/Asphalt/Concrete[d]							
	9	Undetermined[d]							
TSF-07	1	Personal protective equipment							
WRRTF-01	20,070	Silty clay[e]		54	2,360	20		0.542	
D&D	72	Rubble (concrete, metal, building materials)[f]							

a. As per C. Bean and the WAG 5 ROD

b. As per C. Bean and the WAG 10 Decision Document Package

c. As per D. Wiggins and the WAG 4 ROD

d. As per C. Bean and the INEEL Integrated Waste Tracking System (IWTS)

e. As per C. Bean and the WAG 1 ROD

f. As per DOE/ID-10803 (Doornbos 2000).

Portland cement is made up of four main compounds: tricalcium silicate ($3CaO{\bullet}SiO_2$), dicalcium silicate ($2CaO{\bullet}SiO_2$), tricalcium aluminate ($3CaO{\bullet}Al_2O_3$), and a tetra-calcium aluminoferrite ($4CaO{\bullet}Al_2O_3{\bullet}Fe_2O_3$). In an abbreviated notation differing from the normal atomic symbols, these compounds are designated as C_3S, C_2S, C_3A and C_3AF, where C stands for calcium oxide (lime), S for silica, A for alumina, and F for iron oxide. Small amounts of uncombined lime and magnesia also are present, along with alkalis and minor amounts of other elements. The composition of Portland cements falls within the range of 60 to 67 % lime, 19 to 25 % silica, 3 to 8 % alumina, and 0.3 to 6 % iron oxide together with 1 to 3 % sulphur trioxide, derived mainly from the added gypsum, 0.5 to 5 % magnesia, and 0.3 to 1.3 % alkalies. Titanium oxide is usually present to the extent of 0.1 to 0.4 %. Manganese oxide is usually present only in small amounts except when blast-furnace slag is used as a raw material. Then it may rise to 1 %, giving the cement a brownish tinge rather than the normal grey color. A typical mineral composition of ASTM Type I Portland Cement is shown in Table 4.

Table 4. Mineral composition of Type I Portland cement (typical).

Component	Weight %
$3CaO \bullet SiO_2$	45
$2CaO \bullet SiO_2$	27
$3CaO \bullet Al_2O_3$	11
$4CaO \bullet Al_2O_3 \bullet Fe_2O_3$	8
Free CaO	5
$CaSO_4$	3.1

Flyash has a typical composition as shown in Table 5. The shape, fineness, particle-size distribution, density, and composition of flyash particles influence the properties of end use products. Flyash produced at different power plants or at one plant with different coal sources may have different colors. In addition, particle size and shape characteristics of flyash are dependent upon the source and uniformity of the coal, the degree of pulverization before burning, and the type of collection system used. Rapid cooling of the ash from the molten state as it leaves the flame causes flyash to be predominantly noncrystalline (glassy) with minor amounts of crystalline constituents, such as mullite, quartz, magnetite (or ferrel spinel), and hematite. Other constituents which may be present in high-calcium flyash include periclase, anhydrite, lime, alkali sulfate, melilite, merwinite, nepheline, sodalite, C_3S, and C_2A.

Table 5. Typical Flyash Composition (wt %).

Component	Class F	Class C
SiO_2	35	35
Al_2O_3	20	20
Fe_2O_3	6	6
$SiO_2 + Al_2O_3 + Fe_2O_3$	70 min	50 min
SO_3	5 max	5 max
CaO	5	15
MgO	5 max	5 max
H_2O	3 max	3 max
Alkali as Na_2O	1.5 max	1.5 max

Table 6 lists the typical chemical composition of blast furnace slag (BFS). The chemical compositions shown are in general applicable to all types of slag. When ground to the proper fineness, the chemical composition and glassy (noncrystalline) nature of vitrified slags are such that when combined with water, these vitrified slags react to form cementitious hydration products. The magnitude of these cementitious reactions depends on the chemical composition, glass content, and fineness of the slag. The chemical reaction between BFS and water is slow, but it is greatly enhanced by the presence of calcium hydroxide, alkali, and gypsum ($CaSO_4$).

Table 6. Typical composition of blast furnace slag (wt %).

Constituent	Mean %	Range %
Calcium Oxide (CaO)	39	34-43
Silicon Dioxide (SiO_2)	36	27-38
Aluminum Oxide (Al_2O_3)	10	7-12
Magnesium Oxide (MgO)	12	7-15
Iron (FeO or Fe_2O_3)	0.5	0.2-1.6
Manganese Oxide (MnO)	0.44	0.15-0.76
Sulfur (S)	1.4	1-1.9

Because of these cementitious properties, BFS can be used as a supplementary cementitious material either by premixing the slag with Portland cement to produce a blended cement (during the cement production process), or by adding the slag to Portland cement as a mineral admixture. Blast furnace slag is mildly alkaline and exhibits a pH in solution in the range of 8 to 10. Although blast furnace slag contains a small component of elemental sulfur (1 to 2 %), the leachate tends to be slightly alkaline and does not present a corrosion risk to steel in pilings or to steel embedded in concrete made with blast furnace slag cement or aggregates.

The basic hydration chemical reactions that occur in cement reactions include:

$$3CaO \bullet Al_2O_3 + 6H_2O \rightarrow 3CaO \bullet Al_2O_3 \bullet 6H_2O \quad \Delta H = 207\, cal/g$$

$$3CaO \bullet Al_2O_3 + 3CaSO_4 + 32H_2O \rightarrow (CaO)_6 \bullet Al_2O_3 \bullet (SO_4)_3 \bullet 32H_2O \quad \Delta H = 347\, cal/g$$

$$2(3CaO \bullet SiO_2) + 6H_2O \rightarrow 3CaO \bullet 2SiO_2 \bullet 3H_2O + 3(CaO \bullet H_2O) \quad \Delta H = 120\, cal/g$$

$$2(2CaO \bullet SiO_2) + 4H_2O \rightarrow 3CaO \bullet 2SiO_2 \bullet 3H_2O + CaO \bullet H_2O \quad \Delta H = 62\, cal/g$$

$$CaO + H_2O \rightarrow CaO \bullet H_2O \quad \Delta H = 279\, cal/g \ .$$

Proposed cement reactions with waste metals include addition, substitution, formation of new compounds, and multiple mechanisms. The addition and substitution reactions are shown below (using lead as an example metal):

$$xCaO \bullet SiO_2 \bullet H_2O + Pb = Pb \bullet xCaO \bullet SiO_2 \bullet H_2O$$

$$xCaO \bullet SiO_2 \bullet H_2O + xPb = xPbO \bullet SiO_2 \bullet H_2O + xCa \ .$$

4.2.3 Soils

Soil type may impact treatability study results as soils may contain a diverse combination of clay, silt, sand, rock, and natural organic compounds (e.g., humus). The interaction of metals within the soil matrix is complex and difficult to predict. It is known that naturally occurring clays have the ability to provide adsorption and weak ion exchange sites for metals, including lead, mercury and the other contaminants of concern. Additionally, these metals may be dispersed on sites throughout the porous structure within the clay. For cement to stabilize the hazardous metals, these metals must be desorbed from the clay and participate in the cement hydration reactions (cementitious reactions). However, it is questionable whether desorption will take place into the alkaline environment provided by cement. Subjecting this clay, with its sorbed metals, to the acidic solution of the TCLP would likely result in leaching of these metals. This argument is used to justify choosing a soil with a high clay content as a worst case soil type. Evidence to support this argument includes results from treatability studies conducted on INEEL mixed wastes (Gering 1993), wherein high clay content wastes leached hazardous metals at higher than expected levels.

Although soils may be classified as high clay content, they may have a significant fraction of other soil material such as sand or silt. This is demonstrated in Figure 1 where clay soils may actually contain in excess of 40% non-clay material.

The targeted CERCLA clean-up sites are located within three general areas at the INEEL, namely Test Area North (contains WRRTF site), the Power Burst Facility (contains ARA site), and Central and the Test Reactor Area (contains the CFA and CPP sites). Soils from each of these three areas will be procured and a surrogate waste material prepared from each. As shown in Table 7, soils within these are typically of high clay content material. When soils are procured to prepare the surrogates, preference will be given to material with a high clay content.

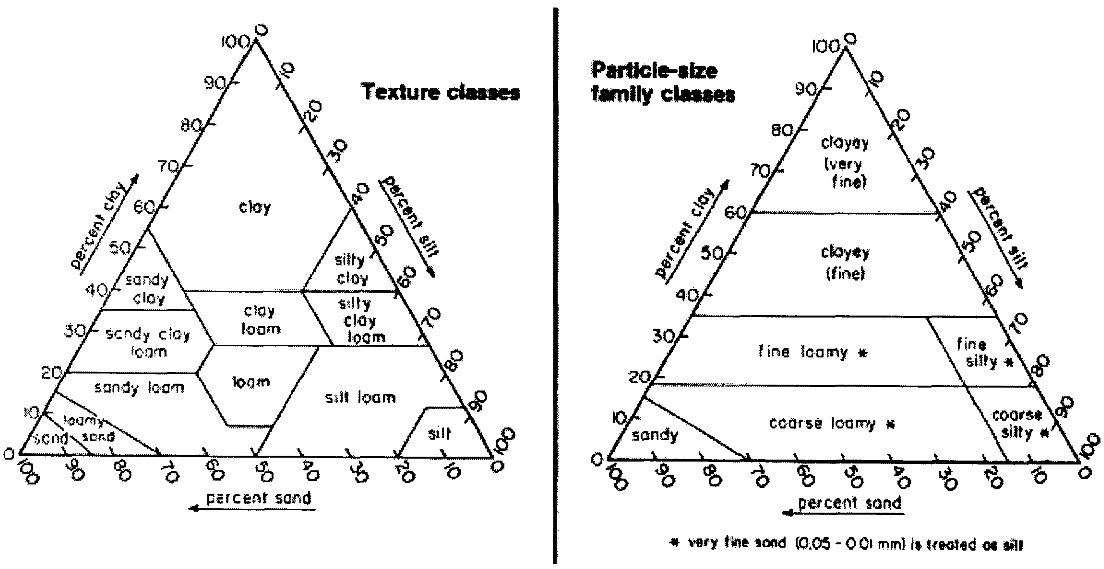

Figure 1. Triangular diagrams depicting soil classification.

Table 7. Site soil matrix information.

WAG	Site	Volume, yd^3	Matrix	Site Description	Reference
5	ARA-12	2,000	Sandy silty clay with rock pieces.	Unlined surface impoundment (370 x 150 ft). Natural depression used to dispose of low-level waste and facility runoff.	Craig Bean (Geotec. Engineer 6-9941); WAG 5 ROD pp 59-63
10	BORAX-01	11,110	Significant imported gravel in an area of silty clay soil	Site of leach pond for the boiling water reactor experiment (BORAX). Dimensions: 20 x 90 ft. Feed included: low-level rad liquid, nonrad cooling tower, H_2SO_4, NaOH, HBO2.	Craig Bean (Geotec. Engineer 6-9941); WAG 10 Track 1 sites: Decision Doc package. DOC ID: 5757 ppl-5
4	CFA-04	800	Rocky soil with a small percentage of calcine	Shallow unlined surface depression (500 x 150 ft). Basalt outcrops are present. Primary discharged: 100 yd^3 Hg contaminated calcine & liquid effluent from calcine laboratory	Debbie Wiggins (WAG 4 Project Engineer 6-9989); WAG 4 ROD pp.8-1 to 8-5
3	CPP-92	1,197	Soil (10%>.75"), (.75"<40%>.25"), (.25"<40%>.75 m), (10%<.75 m)	584 (2 x 4 x 8-ft) boxes + 5 (4 x 4 x 8-ft) boxes [Assumption: Boxes are 85% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		3	Metal	1 (4 x 4 x 8-ft) box Assumption: Box is 60% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		114	Concrete	40 (4 x 4 x 8-ft) boxes Assumption: Boxes are 60% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		51	Soil/Asphalt/Concrete	18 (4 x 4 x 8-ft) boxes Assumption: Boxes are 60% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
3	CPP-98	30	Soil (10%>.75"), (.75"<40%>.25"), (.25"<40%>.75 m), (10%<.75 m)	17 (2 x 4 x 8-ft) boxes [Assumption: Boxes are 85% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		209	Wood / Nails / Bolts	98 (4 x 4 x 8-ft) box Assumption: Box is 45% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		6	Metal	2 (4 x 4 x 8-ft)boxes Assumption: Boxes are 60% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		3	Undetermined	1 (4 x 4 x 8-ft)boxes [Assumption: Boxes are 60% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
3	CPP-99	30	Soil (10%>.75"), (.75"<40%>.25"), (.25"<40%>.75 m), (10%<.75 m)	15 (2 x 4 x 8-ft) boxes Assumption: Boxes are 85% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		2	Wood / Nails / Bolts	1 (4 x 4 x 8-ft) box Assumption: Box is 45% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		11	Metal	5 (4 x 4 x 8-ft) boxes [Assumption: Boxes are 45% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		62	Concrete	29 (4 x 4 x 8-ft) boxes [Assumption: Boxes are 45% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		12	Soil/Asphalt/Concrete	5 (4 x 4 x 8-ft) boxes [Assumption: Boxes are 50% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
		9	Undetermined	4 (4 x 4 x 8-ft) boxes [Assumption: Boxes are 50% full]	Craig Bean (Geotec. Engineer 6-9941) ; IWTS
D&D&D		56	Rubble (concrete, metal, building materials)	Mixed Low-Level Waste (MLLW)	CWID document DOE/1D 10803 pp4-3 to 4-8
		16		Hazardous Waste (HW)	
1	TSF-07	PPE			
1	WRRTF-01	20,070	Silty clay	Four burn pits used for open burning of construction debris. Total Dimensions: 400 x 165-ft. Covered with 1/2 to 9 ft of clean soil.	Craig Bean (Geotec. Engineer 6-9941) ; WAG 1 ROD pp 9-1 to 9-8 section II

4.2.4 Surrogate Testing

Surrogates will be manufactured by spiking representative soils with the contaminants (soluble species) shown in Table 3. The TCLP will be conducted on the untreated matrix and stabilized material in accordance with SW-846 Method 1311 (U.S. EPA 1986a). The success criterion will be if the TCLP on the treated surrogate is less than the LDR. The reagents chosen are based on those known to be effective for the metals present (Conner 1990).

The BFS is used to help form insoluble metal sulfides as the slag contains a small fraction of available sulfur. Soluble sulfide, added as sodium sulfide, is used as pretreatment for mercury, and also produces insoluble or sparingly soluble compounds with other toxic metals. Also, it may be required to include an organic fixating additive to prevent organic compounds from interfering with reactions. The reagents consist of the following:

- Type ASTM I Portland cement (PC)
- Class F Flyash
- Blast Furnace Slag (BFS)
- Na_2S for mercury fixation and stabilization of other metals
- Organic fixating additive (e.g., organo-clay, modified organophillic clay, granulated activated carbon, polyester).

The estimated number of surrogates is based on the following:

- One TCLP on the untreated, spiked surrogate waste sample
- Four CFS formulations
- Two water contents, 20 or 30%
- Three soil types
- Three WLs.

Every fifth sample will be run in duplicate. There will be two WLs, 65 and 80 or 65 and 50; and three soil types for a total surrogate sample count of 1.2 x (3 x 4 x 2 x 3) + 3= 75

The WL is defined (dry basis) as:

$$WL \equiv \frac{wt.\,waste}{wt.\,waste + wt.\,reagent} \quad .$$

A golden mean section[1] (or rule of 2 that is slower to converge) strategy will be used to help minimize the sampling/analysis (Rudd 1968). For soils, it is believed that the range of WLs should be 40-90%. The midpoint of this range is 65. For the testing less than 65, the first golden section is:

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1. The golden section is derived from ratios of successive Fibonacci numbers, 0,1,1,2,3,5,8,13,21,….., 13/21 = 0.619 and is believed to be an optimal search method.

$$GSL_1 = 65 - \frac{65-40}{1.62} = 50$$

and for above 65:

$$GSU_1 = 65 + \frac{65-40}{1.62} = 80 \ .$$

The second golden section is:

$$GSL_2 = 50 - \frac{50-40}{1.62} = 44 \ .$$

There are specific minimum values that can be assigned to CFS formulation reagents. For mercury, it is recommended to not exceed 1.2 times the stoichiometric amount of sulfide (Conner 1990) in a well-mixed system. Therefore, the amount of sulfide needed for fixating Hg for CFA-04 is:

$$\frac{440 mg/kg}{200 mg/mmole} * 1.2 * 78 mg/mmole = 206 \ mg\, Na_2S/kg \ .$$

However, note that lead, chrome, and cadmium also compete for sulfide. (Calcium may also form compounds with sulfide under the proposed treatment scheme). Assuming this reagent will also be consumed by the other toxic metals, additional amounts are needed within each formulation. Based on the surrogate composition given in Table 8, there are 40.24 mmol toxic metals per kg waste, yielding a sulfide requirement of 47.89 mmol, accounting for reaction stoichiometry. At 20% excess, this amount equates to 4.36 g Na_2S/kg, representing the minimum dose of sodium sulfide that would be required in a well-mixed system that has no other sulfide consumers. Due to nonideal mixing and the likelihood of other sulfide sinks, it is recommended that the Na_2S dose be increased to 100% excess, or 7.28 g Na_2S/kg waste. A final comment should be made about the administration of Na_2S. It is recommended that, if possible, the sulfide-bearing compound be added to the mixture comprised of the waste and added water, and be allowed to mix for at least 15 minutes before the addition of PC, flyash, or BSF.

4.2.4.1 Water Addition. Water is a crucial component of treatment methods based on hydraulic binders because it facilitates aqueous phase stabilization reactions, promotes more intimate mixing between waste particles and stabilization reagents, and provides adequate waters of hydration that are required for the hydrated cementitious species that form as the concrete cures.

The amount of water from the soil available for cement reactions is dependent on pore volume and bulk density (also the hydrophillic propensity of cementitious material and silt/clay) and is equal to the quantity:

$$\frac{\varepsilon * fr.Sat * 1kg/L}{\rho_B}$$

$$\varepsilon = 1 - \frac{\rho_B}{\rho_p}$$

Where

ρ_B = Soil bulk density (kg/L)

ρ_P = Soil particle density (kg/L).

The water used during actual stabilization will depend on the dust control water sprayed that is an uncertainty. For the surrogate testing, relatively dry soil will be used with water added to meet the water to cement (W/C) ratio required and workability of the wet CFS formulation. The total amount of water present impacts permeability, the amount of unreacted cement, air voids, and bleed water.

4.2.4.2 Paint Filter Testing. Stabilized samples will be tested with Method 9095A, the paint filter test.

4.2.4.3 Surrogate Composition. The surrogate will consist of the highest concentrations from Table 3 (twice the metal concentration for conservative approach) by spiking the representative soil (on a dry basis) with the metals and organic compounds. Table 8 provides the composition of the surrogate. The amount of water residing in the solid constituents will be accounted for in the final treatment formulations. The three soil types are from Test Area North, the Test Reactor Area, and the Power Burst Facility.

Table 8. Surrogate composition.

Component	Amount
Ag	600 mg/kg
Cd	240 mg/kg
Cr	1900 mg/kg
Hg	880 mg/kg
Pb	6700 mg/kg
Toluene[b]	98 mg/kg
Benzene	5 mg/kg
Carbon Disulfide	0.14 mg/kg
Tetrachloroethylene	0.056 mg/kg
Methylene Chloride	0.29 mg/kg
TCE	0.2 mg/kg
1,1,1-TCA	0.033 mg/kg
1,1,2-TCA	0.009 mg/kg
Chlorobenzene	1.2 mg/kg

a. Silt and clay types should be representative of the WAG soils.

b. Organic compounds are the highest from the CWID database from Preussner (2000).

4.2.4.4 CFS Surrogate Formulations. The CFS formulations to be used in the treatability studies are shown in Tables 9, 10, and 11 for the midpoint and the first two golden sections. The test starts with Portland cement, then uses 6:1 PC/Flyash per Raivo (2000). The third formulation uses 6:1 flyash and 10:1 BFS. The Na_2S is a mercury scavenger, i.e., if the BFS does not fix mercury, the Na_2S is added. The Na_2S is used as a pretreatment; it is added to the (waste + water) portion before mixing with the other reagents. Finally, the amount of added water will be varied within the CFS formulations to achieve total water contents of 20 and 30 wt%. The amounts of water shown in Tables 9 to 11 provide 20 wt% water in the formulation. For 30 wt% water, these values will be adjusted to 66 g in Table 9, 85.8 g in Table 10, and 53.6 g in Table 11.

Surrogate Test Plan Strategy. The overall object of the tests is to obtain a single baseline recipe that delivers an end product that meets the ICDF WAC. This will include meeting LDRs for hazardous metals andpassing the paint filter. Additionally, a friable end product is desired (but not a current requirement) to allow easier materials handling during full-scale operations.

To begin the tests, the 4 recipes in Table 9 (corresponds to 65wt% waste loading) will be prepared. The 4 samples will be analyzed by the TCLP and the paint filter test. This is a decision point as the results of these analyses will determine the next set of tests as outlined below.

- *Successful results at 65wt% waste loading:* All formulations that are successful will be used at 80wt% waste loadings (corresponds to the formulations in Table 11). These 80wt% samples will be analyzed for performance and all successful formulations will be tested at progressively higher waste loading until failure. Tables for formulations beyond 80wt% waste loadings were not presented in the text. If all 80% formulations fail, intermediate loadings between 65 and 80% would be used to fine-tune the formulation.
- *Unsuccessful results at 65wt% waste loading:* If all formulations are unsuccessful at 65wt% waste loadings, the mode of failure will guide the next set of tests. If failure is by the paint filter test, the water in the formulation will be reduced and the 65wt% waste loading tests will be repeated. If failure by TCLP, the waste loading will be reduced to 50wt% and the formulations of Table 10 will be used. If all formulations fail at 50%, the mode of failure and any trends in these failures will be used to adjust the formulations. This may require the use of additional reagents.

An exhaustive list of the potential failure modes and consequent adjustments has not been developed. Such a list would be exceedingly long and are viewed as being of limited benefit. Failure trends found during the tests will be used to guide adjustments as necessary. For example, water content will likely be a significant parameter in the "set" of the final waste form. Although the stabilized waste may meet the TCLP and other primary disposal criteria, the water content may be varied to obtain the desired friable end product.

4.2.4.5

Table 9. CFS surrogate formulations for midpoint (65%).

	Waste Loading wt%	Surrogate Amount, g	Portland Cement, g	Flyash, g	BFS, g	Sulfide, g	Water, g
Recipe #1	64.94	100	54	0	0	0	38.7
Recipe #2	65.08	100	46	7.67	0	0	38.7
Recipe #3	65.01	100	42.5	7.08	4.25	0	38.7
Recipe #4	64.97	100	42	7.00	4.2	0.728	38.7

Table 10. CFS surrogate formulations for ≈ 50% WL.

	Waste Loading wt%	Surrogate Amount, g	Portland Cement, g	Flyash, g	BFS, g	Sulfide, g	Water, g
Recipe #1	50.00	100	100	0	0	0	50
Recipe #2	50.06	100	85.5	14.25	0	0	50
Recipe #3	50.14	100	78.5	13.08	7.85	0	50
Recipe #4	50.12	100	78	13.0	7.8	0.728	50

Table 11. CFS surrogate formulations for ≈ 80% WL.

	Waste Loading wt%	Surrogate Amount, g	Portland Cement, g	Flyash, g	BFS, g	Sulfide, g	Water, g
Recipe #1	80.00	100	25	0	0	0	31.3
Recipe #2	79.95	100	21.5	3.58	0	0	31.3
Recipe #3	80.19	100	19.5	3.25	1.95	0	31.3
Recipe #4	80.13	100	19	3.17	1.9	0.728	31.3

Table 12. Sample results table.

CFS recipe #1, WL 65%	Untreated mg/kg (TCLP)	Treated mg/kg (TCLP)
Ag		
Cd		
Cr		
Hg		
Pb		

4.3 Quality Assurance and Quality Control

The QC/QA requirements for this project are established in the following two environmental restoration plans:

Implementing Project Management Plan for the Idaho National Engineering and Environmental Laboratory Remediation Program(U.S. EPA 1988).

Quality Assurance Project Plan for Waste Area Groups 1, 2, 3, 4, 5, 6, 7, 10 and Inactive Sites (DOE/ID 1997).

In accordance to company-wide management control procedure (MCP)-540 "Graded Approach and Quality Level," OU 3-13 characterization and treatability study activities are a Quality Level 3. Field logbooks, chain-of-custody forms, Environmental Restoration Information System (ERIS) data files, and data limitations and validation reports will be rigorously controlled as defined by the INEEL Sample Management Office (SMO) and outlined in the applicable technical procedures (TPRs) and MCPs specified throughout this work plan. Definitive data (U.S. EPA 1993) will be produced including all field sampling and toxicity characteristic, and paint filter analyses results. These analytical tests will be performed in accordance with the applicable EPA reference methods, the Field Sampling Plan, and the Task Order Statement of Work (TOS). Quality Control samples must meet the minimum requirements stated in the Quality Assurance Project Plan (QAPjP) (DOE-ID 1997). These samples will also be included in the TOS and the SAP tables in the FSP. The analytical data packages submitted will be validated to validation level "B," as defined by the QAPjP.

5. EQUIPMENT AND MATERIALS

5.1 Surrogates

5.1.1 Sample Preparation

Samples will be prepared by spiking the soil with the RCRA metals and organic compounds listed in Section 5.3 to obtain the concentrations in Table 8. The samples will be well mixed to ensure the components are evenly distributed.

5.1.2 Stabilization

The solidification agent will be dry mixed and stored before being added to the waste form so that it will be ready when the samples are ready. There will be two samples prepared for each CFS# from two separate batches made to the same specifications. Once the surrogate samples are prepared, they will be put in storage until the laboratory is prepared to solidify the sample. Samples will be mixed either by hand or with a rotary mixer and then poured or scooped into molds. The spiked soil will be placed in the mixing vessel (see equipment list in Section 5.3). The top of the mixing vessel will be covered to prevent spills during mixing. Efforts will be made to minimize contact between the mixture and its surroundings. Drip pans and other precautions will be used to minimize contact. Where practical, equipment will be rinsed; however, if this results in the generation of too much waste or it is impractical, the vessels will be disposed of without rinsing.

Several years of mixed waste treatability studies performed for INEEL's Waste Reduction Operations Complex have yielded preferred procedures for mixing cement mixtures (Gering and Schwendiman 1997). The general recommended procedure is to place the preweighed amount of waste (or surrogate) in the mixing vessel, followed by the total amount of added water. Next, this (waste + water) combination is mixed until the waste becomes an even consistency. At this point, any pretreatment reagents (e.g., Na_2S) can be introduced to the wetted waste as mixing continues. Mixing during pretreatment should be of sufficient duration to allow the pretreatment processes (e.g., redox reactions) to near completion. Next, the hydraulic binders are slowly added as mixing continues.

Standardized solidification test specimens will be prepared in accordance with ASTM C192,[m] (ASTM 1998a). Samples will be poured into 2-in. molds and tamped with a rod to remove air pockets and to get the best compaction. Individual molds will be placed in a curing box and allowed to cure at a controlled temperature between 60 and 80°F until the curing cycle is partially[m] complete. The curing box prevents evaporation and provides some control of the humidity. After 8–12[m] hours, the samples will be removed from their molds.[m]

Once the samples have been removed from the molds, the solidified samples will be tested by the paint filter test for free liquid determination (U.S. EPA 1986b). The TCLP analysis will evaluate the leaching properties of the samples in accordance with Method 1311 (U.S. EPA 1986a).

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m. There are significant variations to ASTM C192 in this procedure as the stabilized surrogates are not intended to be performance grouts. These variations are flagged with this footnote.

5.2 Equipment

- Mixer (drill press, hand drill, or equivalent)
- Mixing attachments
- Spoons or spatulas for transfer of treated mixtures
- Curing box
- Stainless steel beakers (mixing vessels)
- Thermometer
- Balance
- Dry mix storage container
- Dry mix preparation container
- Tamping rods
- Rubber mallet
- Handling tongs
- Waste containers
- Glovebox or hood (for verification testing if required)
- Paint filter Test equipment per Method 9095A (U.S. EPA 1986b)
- TCLP equipment per Method 1311 (U.S. EPA 1986a)

5.3 Materials

- Type I Portland cement
- Class F Flyash
- Blast Furnace Slag (ground, granulated - rapid water quenching)
- Na_2S
- Powdered Activated Carbon
- Water (standard, plant potable water)
- Silty Clay Soil (10 kg)
- $Ag(NO_3)$
- $Cd(NO_3)_2$
- $KCrO_4$
- $Hg(NO_3)_2$
- $Pb(NO_3)_2$
- Benzene
- Chlorobenzene
- Carbon Disulfide
- Methylene Chloride
- 1,1,1-Trichloroethane
- 1,1,2-Trichloroethane
- Tetrachloroethylene
- Trichloroethylene
- Toluene

6. SAMPLING AND ANALYSIS

There may be as many as 80 samples generated from this treatability study. All samples will be analyzed for leachable metals (TCLP), free liquids to determine the success of the treatment process. Analysis of these samples will be coordinated through the Sample Management Office who will provide a Task Order Statement of Work (TOS) and Sampling and Analysis Plan (SAP) for this project. The TOS and SAP will, in accordance with the Quality Assurance Project plan, specify the quality control requirements on the all samples in this study. Sample identification and tracking will be specified in the TOS and SAP and shall conform to MCP-244, "Chain-of Custody, Sample Handling, and Packaging for CERCLA Activities." Samples will be presumed to be hazardous and will shipped in accordance with MCP-2669, "Hazard Material Shipping."

7. DATA MANAGEMENT

Data generated during treatability studies will be managed in accordance with guidelines provided in the *Data Management Plan for the Idaho National Engineering Laboratory Environmental Restoration Program* (INEL 1995a). This plan provides or references procedures and requirements, necessary to develop a database of relevant information that can be readily accessible and accurately maintained. The plan describes the data-flow process, data custodianship, and organizational and individual responsibilities associated with data management. The plan also provides the project file and reporting requirements, and identifies extensive database capability requirements to allow selective data sorting, analysis, formatting, and reporting.

The data management plan provides the necessary requirements for this treatability study. There may, however, be some deviations from the data management plan. Deviations are due to information that is not directly recorded in logbooks or from laboratory data that may not be tracked by the Environmental Restoration Information System (ERIS). For this treatability study, the following tests may result in information considered to be deviations from the Data Management Plan:

- Mixing and Grouting Performance - laboratory data not tracked in ERIS
- Paint Filter Test - laboratory data not tracked in ERIS

In each of the above-mentioned cases, the data and information may be placed in Information Repository and Document Control, if it is not tracked in ERIS. Additionally, hard copies of the raw data and test results will be summarized in engineering design files (EDFs) following each scheduled test. Specific DQOs and data validation requirements will be specified in the test plans. Program Requirements Document 111, "Records and Forms Management," will assist in ensuring that information is available when needed, protected as appropriate, and properly dispositioned. In addition, a number of Management Control Procedures (MCPs) will be invoked during this Treatability Study (TS) process for activities performed at the INEEL. The primary MCPs that will be used are as follows:

- MCP-227, "Sampling and Analysis Process for EM-Funded Activities"
- MCP-230, "Environmental Restoration Document Control Center Interface"
- MCP-231, "Logbooks"
- MCP-242, "Obtaining Laboratory Services for EM-Funded Activities"
- MCP-244, "Chain-of-Custody, Sample Handling, and Packaging for Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Activities"
- MCP-452, "Treatability Studies"
- MCP-2864, "Sample Management."

8. DATA ANALYSIS AND INTERPRETATION

Upon completing the TS, the data will be summarized and evaluated to determine the validity of the data and to assess the performance of the stabilization process. To accomplish this goal, results will be reduced to a useful form in accordance with applicable data uses, including specifically:

- Characteristic metals immobilization
- Free liquid determination by the paint filter test

The data will be both qualitative and quantitative. The qualitative data will include photographic records of the bench-scale testing, visual observations, logbook entries, descriptions, etc. The quantitative data will include timing of events, measurements of the amount of materials used, chemical concentration measurements, physical measurements, mixing parameters, and laboratory analyses.

Data produced from testing will be reported as described in Section 7. Complete data from the TS will be summarized in Engineering Design Files (EDFs) by project personnel upon completion of the laboratory work. The EDFs will provide the key information needed for complete data analysis and interpretation in the TS report. In addition to the analytical data collected during the study, data packages will also contain relevant observations of key parameters and unknowns encountered during the testing.

Test results are to be interpreted in the context of the formulation's effectiveness, implementability, and costs of the procedures to perform at full-scale operation.

9. HEALTH AND SAFETY

The TS will be conducted onsite—the particular laboratory has not yet been identified. All site laboratories require an Independent Hazard Review (IHR) of all new activities, as per MCP-3571 "Independent Hazard Review." This review is a thorough investigation of the planned activities and requires the approval of the environmental, health, and safety professionals assigned to the particular laboratory. Specifically, this includes approval by Industrial Hygiene, Fire Safety, Radiological Engineering, Waste Generator Services, Laboratory Lead Chemist (custodian) and finally, the Environmental, Safety and Health Laboratory Manager. The documentation submitted for IHR approval must include the following:

- Identify and detail the hazardous and radionuclides levels associated with all chemicals and materials planned for use
- Identify and evaluate risks in doing the work
- Estimate the types and quantities of wastes generated
- Identify the type of PPE.

In addition, a hazard mitigation plan must be included in the IHR package, which identifies the work site control measures (radiological buffer areas, radiation area, etc.), medical surveillance requirements for personnel, personnel monitoring requirements, and the PPE requirements.

10. RESIDUALS MANAGEMENT

All onsite laboratory activities require submittal and approval of Independent Hazard Review (IHR) documentation before beginning any laboratory work. A section of the IHR documentation is dedicated to determining the types and amounts of wastes that will be generated from the proposed activity. The IHR documentation must be reviewed and approved by the laboratory's Waste Generator Services representative to insure that all wastes generated are properly tracked, stored, and disposed of.

The treatability studies discussed herein will be performed on surrogate wastes containing heavy metals- no radioactive materials will be used. Wastes generated as a consequence of this study may include the following:

- Unused, untreated surrogates
- Pretreated wastes, if any
- Stabilized waste forms
- Treatment residues
- Extraction fluids (TCLP)
- Contaminated equipment wash/rinse water
- Contaminated protective clothing and other PPE
- Contaminated sampling materials and debris.

10.1 Waste from Surrogate Tests

The tests on surrogate waste will use soils spiked with leachable heavy metals; therefore, there is a potential for generating toxic metal-bearing hazardous waste. The surrogates may also be spiked with organics; however, the amount of organic solvents added during surrogate preparation will be controlled so that the resulting material would not be hazardous for organics, even without treatment. Through careful planning, the amount of hazardous waste generated will be minimal. All surrogate material will be consumed during the stabilization tests, i.e., all surrogate material will undergo stabilization. No radioactive material will be used during surrogate testing. Incidental lab waste that is not hazardous will be disposed of as common waste. Waste identified as hazardous (treated surrogate that fails TCLP) will be entered into the laboratory's satellite accumulation area and its disposal coordinated with Waste Generator Services.

10.2 Hazardous Waste Determination

The TCLP analyses will either be performed on-site at the INTEC analytical laboratory or by an off-site subcontractor. Subcontracting for analytical services will be conducted through the Sample Management Office, which identifies suitable subcontractor laboratories. Several of these subcontractors dispose of TCLP residues; however, if a subcontractor is selected that does not have disposal capabilities, the residues will be returned to the treatability laboratory. Storage and disposal of these residues will be coordinated with the laboratory's Waste Generator Services representative. The analytical laboratory at INTEC has disposal methods in place for TCLP residues.

11. COMMUNITY RELATIONS

The community relations task is designed to ensure community understanding of actions taken during the CERCLA remediation action and to obtain community input on the remediation program. Community relations area an integral part of any CERCLA action, regardless of whether or not the action is at a federal facility. At the INEEL, all CERCLA actions will be subject to both CERCLA and National Environmental Policy Act community involvement requirements. The INEEL public affairs group has prepared a programmatic Environmental Restoration Program Community Relations Plan (CRP) (INEEL, 1995b) that covers the Remedial Investigation/Feasibility Study process at the INEEL. This CRP was issued as a DOE document representing "the process established by mutual agreement between the DOE, EPA, and State of Idaho to address ER concerns at the INEEL." The CRP will guide the actions taken to ensure appropriate public involvement in agency decision-making.

12. REPORTS

During the course of the TS, open lines of communication are essential to ensure a smooth and accurate flow of information to all parties directly or indirectly involved with the project. The TS work package manager is responsible to ensure that pertinent information is timely disseminated to interested parties, using informal project meetings or conference calls and notes, as well as more formal written reports.

The organization performing the TS will document activities by preparing the following reports:

- Brief letter status reports documenting the preliminary performance of CFS formulations, including any trending information and need to alter formulations.
- At the completion of the surrogate testing, Engineering Design Files will be prepared that will detail the methodologies used, summarize and interpret experimental data, and provide recommendations for CFS formulations for use at the SSSTF.

13. SCHEDULE

No schedule has been approved for the treatability study.

14. MANAGEMENT AND STAFFING

No personnel have been committed to this treatability study.

15. BUDGET

The following estimated costs were based on site labor required to support a treatability study, equipment and materials, and off-site analysis of samples.

On-site labor:

Initial document preparation/approval	$ 53,413
Laboratory and support labor	$ 50,414
Data validation/reporting	$ 18,391
Equipment and Material:	$ 5,538
Subcontractor (TCLP analysis, 80 samples):	$ 44,306
TOTAL COST	$ 172,062

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MCP-231, "Logbooks"

MCP-241, "Preparation of Characterization Plans"

MCP-242, "Obtaining Laboratory Services for EM-Funded Activities"

MCP-244, "Chain-of-Custody, Sample Handling, and Packaging for CERCLA Activities"

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